

Application Note

Keywords

- Thin films
- Polymers
- Thickness
- Interference pattern

Techniques

- Transmission
- Refraction
- Reflectometry
- Ellipsometry

Applications

- Thin film thickness measurement
- Single and multilayer analysis

Transmission Measurements of Polymer Thin Films

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Transmission measurements can be used to study the composition, color and even the impurities in a material. When that material is formed into a thin sheet of uniform thickness, transmission measurements can also be used to study its index of refraction or thickness. The key is an optical phenomenon called thin film interference -- the same effect that causes soap bubbles to shimmer with a rainbow of colors when illuminated. When using this method, it is important to select the optimum wavelength range of light for the material and thickness under study. In this feasibility study, thin polymer sheets were analyzed using the NIRQuest512 spectrometer to see if a clear interference pattern could be obtained.

Introduction

Thin film interference occurs when light reflects from two parallel surfaces and those light rays interfere constructively and destructively. The resulting transmission spectrum then has a sinusoidal pattern of interference called "fringes" superimposed on it, as seen in Figure 1.

When light is incident at 90° (as in transmission), the maxima will appear at:

$$2nd = (m - \frac{1}{2}) \lambda$$

where n is the index of refraction of the material, d is its thickness, m is the fringe number, and λ is the wavelength of light being used. When the wavelengths at which the maxima occur are plotted versus fringe numbers starting at x (then $x+1$, $x+2$ and so on), a straight line emerges with a slope

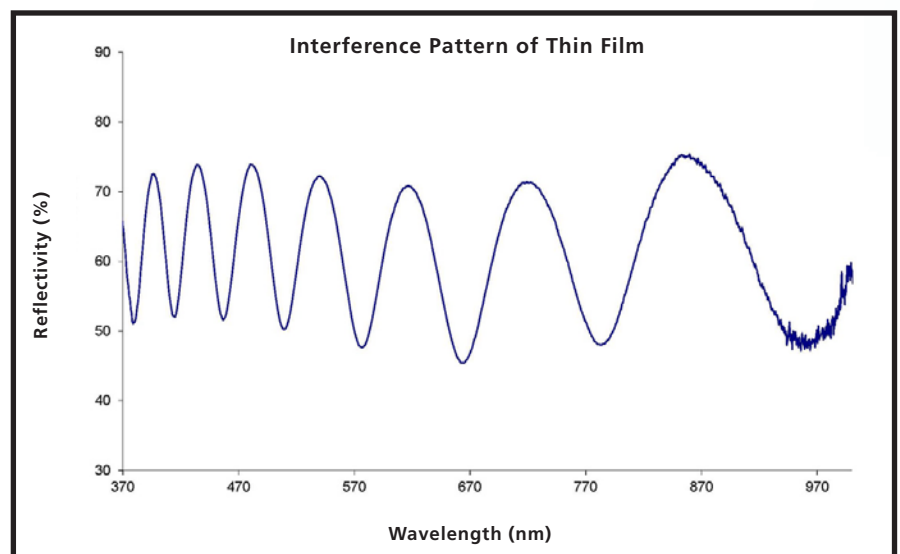


Figure 1: Thin film interference is demonstrated in a sinusoidal pattern in the transmission spectrum.

equal to $1/(2nd)$, as seen in Figure 2. The thickness can then easily be calculated if the index of refraction is known, or vice versa. Even if the index of refraction is unknown, this method can still be used for process control, where limits can be set on the slope value based on a group of known acceptable samples.

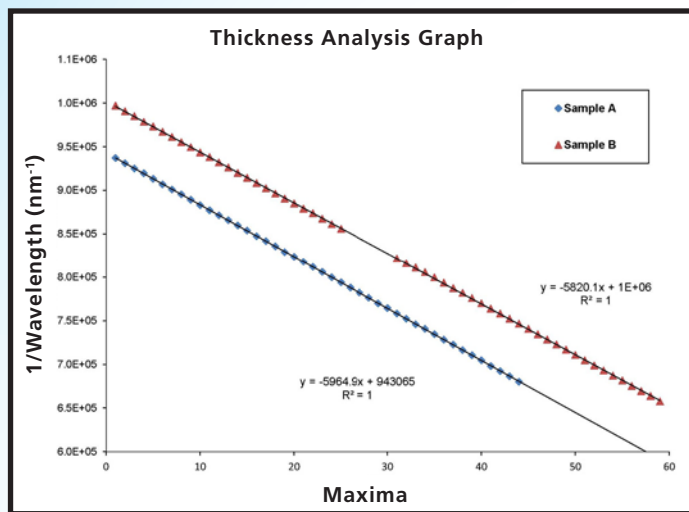


Figure 2: Wavelengths at which the maxima occur are plotted versus interference fringes to help calculate thickness.

For best results, a wavelength of light should be chosen that allows you to see at least 5-10 fringes in the transmission spectrum across the spectrometer's wavelength range. Each fringe should have enough data points to allow the maximum to be easily identified or interpolated by drawing a smooth line through the data. Maxima will be closer together at shorter wavelengths, so if you find that you have too few fringes, you may want to try a shorter wavelength range. Also, you can plot the minima as a separate line to verify your results, as it should yield the same slope.

Experimental Conditions

Three translucent polymer film samples were examined in transmission over the range 850-1700 nm using a NIRQuest512 spectrometer configured with a 25 μm slit, yielding an optical resolution of ~ 4.0 nm (FWHM). A high power tungsten halogen lamp was chosen as the light source, with two 600 μm VIS-NIR fiber assemblies routing light to and from an adjustable collimating lens holder. A high power lamp is warranted when using an NIR spectrometer to keep data acquisition time short, as the InGaAs

detector at the core of the NIRQuest512 is not as sensitive as the silicon detectors used in visible spectrometers. Larger core fibers also help to maximize light throughput.

The polymer films were affixed directly to an arm of the collimating lens holder, ensuring that the films remained flat against the arm. Transmission spectra were acquired in software using a 50 ms integration time, averaging 10 scans, with boxcar (smoothing) set to 0. Averaging is preferred over boxcar smoothing in this case in order to preserve the resolution of the spectrometer.

When aligning the samples for measurement, it is important to first align the collimating lenses installed in the transmission stage. Each lens is in two parts – an outer sleeve that screws into the transmission stage and contains the lens itself, and an inner sleeve that screws onto the fiber. This dual sleeve arrangement allows the distance between the fiber and the lens to be adjusted and then fixed using a set screw. First, the lens on the illumination side should be adjusted so that the light coming from that arm is as collimated as possible. Then, the lens on the receiving fiber side should be adjusted to maximize light throughput while watching the signal level on the spectrometer prior to taking a reference spectrum.

Results

The transmission spectra obtained for the three samples are shown in Figure 3. A flat reference line at 100% is a good indicator that the system has sufficient light and sensitivity to yield low-noise measurements (even at the wavelength extremes of the spectrometer).

Samples A and B show clear, regular interference fringes spaced approximately every 10 nm in the central portion of the wavelength range, with 5-6 data points between maxima. Ideally, there would be 10 or more points per fringe to allow interpolation of the maxima. A smaller slit wouldn't improve this, as it would only increase the optical resolution of the system, not yield more data points (the number of pixels and wavelength range of the spectrometer wouldn't change). As a result, we are

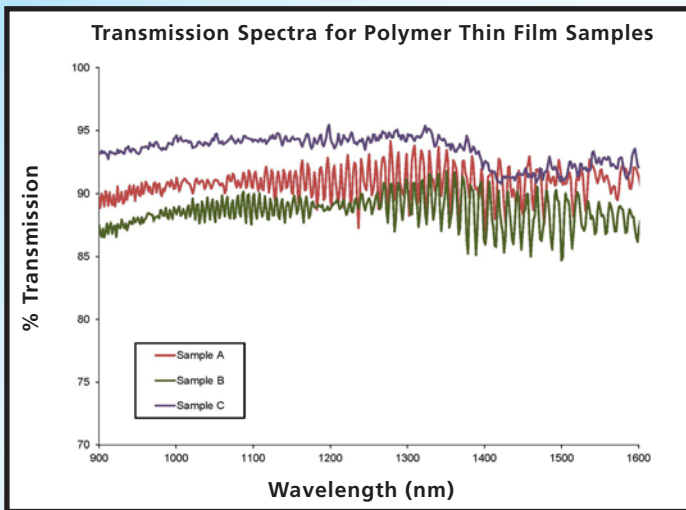


Figure 3: Transmission spectra for polymer thin film samples reveal differences in interference fringes.

really at the limit of how thin a sample this system can measure, but there are enough maxima to be able to make a reasonable guess at each one and through sheer statistical analysis still get a fairly accurate slope as the result. Thicker films would have more widely spaced fringes and be easier to analyze.

Sample C does not show a clear, regular interference spectrum, which could be due to its thickness or index of refraction being too low to show fringes within the resolution limits of the system (remember, maxima occur every $2nd$ in wavelength). Or, it may not be smooth or uniform enough in thickness to yield clear interference fringes, in which case moving to another location on the film could potentially improve the spectrum.

Since the sample can't be altered, the other alternative is to optimize the system. For samples A and B in particular, we would need a smaller wavelength range with the same number of data points. This could be achieved using a higher groove density (lines/mm) grating. The signal level would still be good, since integration time with the test setup was relatively low, and the samples are stationary.

Conclusion

The success of a thin film interference measurement depends heavily on the spectrometer being well-matched to the samples under study. A quick

feasibility measurement with a standard system can easily set you on the right path to choosing the optimal system configuration.

For those who need more from an interference measurement, reflectometry may be the best solution. A system such as the NanoCalc Thin Film Reflectometer takes the next step and fits the spectrum to a well-understood model, provided that the thickness or index of refraction of the layers is known. This method can be much more accurate, and allows analysis of several layers of differing composition, not just a single film.

An ellipsometry system can be even more sensitive. The SpecEI Ellipsometer uses polarized light at a known angle to determine the phase change upon reflection, extracting thickness, index of refraction and absorbance of a sample quickly and easily. 🐼

Contact us today for more information on setting up your spectroscopy system from Ocean Optics.

